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## IV. "On Oxalurate of Ammonia as a Constituent of Human Urine."

By EDWARD SCHUNCK, F.R.S. Received November 15, 1866\*.

When urine is allowed to percolate through animal charcoal in the manner described in the preceding Paper, several organic substances are absorbed and separated by the charcoal in addition to the fatty acid there referred to. The liquid obtained by treating the charcoal with boiling alcohol yields on evaporation a syrupy residue, of which a great part dissolves in water, the fatty acid being left undissolved. The filtered liquid on being again evaporated leaves a brown syrup, among which a quantity of yellowish crystals is formed on standing. On treating the mass with cold alcohol, the syrupy portion, consisting of urinary extractive matter, is removed, the crystals being left undissolved. The latter are filtered off, washed with alcohol, and then dissolved in boiling water. The solution, which has a slightly yellow colour, is evaporated to a small volume, and the crystals, which separate on standing, are pressed between blotting-paper and then dissolved in a little boiling water to which a small quantity of animal charcoal is added. The filtered solution, if tolerably concentrated, becomes on cooling almost solid, from the formation of a quantity of white crystalline needles, which, after the liquid has been drained off, only require drying. The substance as thus prepared consists of pure oxalurate of ammonia, since it is found to possess both the properties and the composition of that salt, as I shall now proceed to show.

The crystals of which it consists are mostly small, and exhibit, even when magnified, few well-defined forms. When a few drops of the watery solution are allowed to evaporate spontaneously on a slip of glass, the residue, when viewed under the microscope, is found to consist mainly of groups of crystals arranged round centres in various irregular forms, the larger ones being composed of prisms, which are acuminate, jagged at the edges, and transversely striated, the smaller ones of needles arranged in star-shaped, double fan-shaped, or circular masses. Occasionally isolated crystals are seen, having the form of rhombic plates, some of which have two of their opposite angles truncated. I have not yet had an opportunity of comparing these forms with those exhibited by the oxalurate of ammonia obtained directly from uric acid. The substance is tolerably soluble in boiling water, but very slightly soluble in boiling alcohol, the little which dissolves in the latter being deposited, on the solution cooling, in fine needles arranged in stars. The watery solution is neutral to test-paper; but on allowing a drop to fall on blue litmus-paper, and exposing the latter to the air for some hours, the spot will appear quite red. The watery solution, on being mixed with hydrochloric or nitric acid, yields a white crystalline deposit (oxaluric acid), which, on being left in contact with the acid liquid, gradually disappears. If nitric acid has been employed and the solution, after

\* Read November 15, 1866: see Abstract, vol. xv. p. 259.

the deposit has dissolved, be spontaneously evaporated, a mass of crystals is left, some of which have the well-known form of nitrate of urea, while the others are prismatic, and consist doubtless of oxalic acid. If the solution, after the addition of any strong acid, be boiled, oxalic acid may after a few moments be detected in it. The watery solution gives no precipitate with chloride of calcium, not even on the addition of ammonia; but on boiling, an abundant precipitation of oxalate of lime takes place. If a tolerably concentrated solution be mixed with chloride of calcium and left to stand, it deposits after some time a quantity of prismatic lustrous crystals, consisting doubtless of oxalurate of lime. The watery solution gives no immediate precipitate with nitrate of silver; but after a few moments it begins to deposit white crystalline needles, which, if the solution was concentrated, increase to such an extent as to fill the whole liquid. These needles are silky in appearance, and do not blacken on exposure to the light, but only become slightly yellow; they dissolve easily in ammonia, but no reduction takes place on boiling the solution. The watery solution of the substance gives with acetate of lead a copious crystalline deposit, and if this be filtered off, the solution yields on standing a crop of small lustrous crystals. These crystals, when examined under the microscope, are found to have very regular forms, consisting of elongated four-sided prisms, with six terminal faces. Whether this form is the same as that of the oxalurate of lead, prepared with acid obtained from the usual source, I cannot say, as I have been unable to find any description of the salt in the books. The watery solution gives no precipitate with perchloride of mercury; but on the addition of chloride of zinc it deposits after some time a quantity of white, hard crystalline grains, which, after being filtered off and washed, are found to contain no chlorine, and on being heated, melt and burn, leaving a white residue of oxide of zinc. If the substance is dissolved in dilute hydrochloric acid, and the solution, after the addition of bichloride of platinum, is evaporated to dryness, the residue on being treated with cold alcohol dissolves partly, a quantity of shining yellow crystals, consisting of chloride of platinum and ammonium, being left undissolved.

Such are the principal reactions of this substance. Its analysis yielded the following results:—

0·6230 grm. lost, on being heated for several hours in the water-bath, 0·0030 grm., or 0·48 per cent., a loss too trifling to be attributed to anything but hygroscopic moisture.

0·3565 grm. of the dry substance gave 0·3150 grm. carbonic acid and 0·1615 grm. water.

0·2605 grm., burnt with soda-lime, gave 1·1425 grm. chloride of platinum and ammonium.

These numbers lead to the formula  $C_6 H_7 N_3 O_8$ , which is that of oxalurate of ammonia, and requires

	Calculation.		Experiment.
C <sub>6</sub> .....	36	24·16	24·09
H <sub>7</sub> .....	7	4·70	5·03
N <sub>3</sub> .....	42	28·19	27·54
O <sub>8</sub> .....	64	42·95	43·34
	149	100·00	100·00

It is therefore certain that the substance obtained by this process is oxalurate of ammonia. These experiments, however, by no means decide the question whether the oxaluric acid exists originally in a free or combined state, since most chemists deny the presence of ready-formed ammonia in urine; and it is quite possible that in my experiments a sufficient quantity of ammonia was formed by the decomposition of urea to saturate the oxaluric acid present. Still I incline to the opinion that the ammonia-salt preexisted in the urine examined, since there were no perceptible indications of decomposition during the percolation of the urine through animal charcoal, a process, indeed, which would rather tend to prevent decomposition than to promote it. The acid reaction of the urine might be urged as an objection to this view; but, on the other hand, it may safely be asserted that we are still in the dark as to the cause of the acid reaction of urine, which may be due to an acid or acids much weaker than oxaluric.

Whether oxaluric acid, either free or combined, is a normal constituent of human urine or not, is a question which may also be raised; but it is one to which I am unable to give a decided reply, as my experiments are not sufficiently numerous for the purpose. I may venture, however, to express my opinion that this acid will be found to be a constituent of the healthy secretion\*. The presence of oxaluric acid in urine had been previously suspected, since the dumb-bell crystals occasionally found among the deposits of oxalate of lime are supposed, by Golding Bird and others, to consist of oxalurate of lime, though the evidence on which this opinion is founded is unsatisfactory, and has been refuted by other observers.

On the other hand, there can be no doubt that the presence of oxaluric acid or its compounds in urine, whether it be an exceptional phenomenon or not, serves to explain, in an easy and satisfactory manner, the formation

\* In a mixture, or any impure product supposed to contain oxaluric acid, I would recommend its detection in the following manner:—The matter, if soluble, should be dissolved in water; but if it is insoluble, in consequence of the presence of some base, a little sulphuric acid should be added to set at liberty the oxaluric acid, after which the solution should be mixed with acetate of lead; and if any precipitate is thereby produced, this must be filtered off and the liquid left to stand, when it deposits small shining crystals if oxaluric acid is present. The residue obtained by evaporation of the mother-liquid of creatine, obtained from urine in the usual manner by means of chloride of zinc, gave, when treated in this way, crystals which could not be distinguished by their form from oxalurate of lead. Oxalurate of silver, distinctly crystallized, can only be obtained from perfectly pure oxaluric acid.

of oxalate of lime so often taking place in the secretion. The appearance of oxalate of lime as a deposit from urine long after its emission has hitherto been a puzzling phenomenon, and the most improbable hypotheses have been resorted to in order to explain it. It has, for instance, been assumed that there exists in the animal economy a tendency to the formation of a soluble triple compound of oxalic acid, lime, and albumen, which, by its decomposition, allows oxalate of lime to crystallize. Then it has been maintained by Rees that uric acid and the urates furnish oxalic acid when the urine containing them is simply heated or boiled; though this statement is questioned by other observers, and it is certain that under ordinary circumstances the conversion of one into the other can only be effected by means of very powerful oxidizing agents such as nitric acid. The attempts which have been made to prove that oxalate of lime may exist ready formed and in a state of solution in the urine are also unsatisfactory, the only known solvent likely to occur naturally being acid phosphate of soda. Were this salt really the means of keeping the oxalate dissolved, the latter would only be deposited when the acid reaction of the urine had disappeared, or had at least somewhat diminished, which is not the case. Now, however, the whole process may be easily explained. Oxaluric acid, as all chemists know, may be considered as a compound of oxalic acid and urea minus water, its composition corresponding to that of oxamic acid. By the action of acids, alkalies, or even water at a high temperature, it is decomposed, yielding oxalic acid and urea. How easily this process of decomposition may be set up in urine when allowed to stand, or even boiled, need not be pointed out. The oxalic acid as soon as formed combines, of course, with the lime which is always present in urine, producing the well-known deposit of oxalate. Those who maintain, with Rees, that oxalate of lime may be produced in the urine after excretion are therefore quite correct, though the phenomenon has hitherto been wrongly interpreted. The conversion of oxaluric into oxalic acid may, however, commence already in the bladder, or even more remote parts of the system, and thus lead to the formation of concretions and calculi. Regarding the origin of the oxaluric acid of urine there can be little doubt. In the animal frame, just as in the laboratory, it must be formed by the oxidation of uric acid, which is its only known source; it may be considered as the vehicle appointed by nature for getting rid of oxalic acid in the least injurious form. Were this acid excreted as such, it would, by combining with lime, produce serious results, which are prevented by the simple expedient of causing it to pass off in a state of intimate union with urea.